JEN-0052-D

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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| Applicant: | Jia et al. | ) | |
| | | ) | Group Art Unit: 1754 |
| Serial No.: | 10/683,750 | ) | |
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| Filed: | October 10, 2003 | ) | Examiner: Paul A. Wartalowicz |
| | | ) | |
| For: | DENTAL RESIN MATERIALS,<br>METHOD OF MANUFACTURE<br>AND USES THEREOF | ) | |

## DECLARATION UNDER 37 CFR 1.132

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Weitao Jia, Ph.D., declares and says that:

1. I am an inventor of the above-referenced U.S. Patent Application No. 10/683,750, herein referred to as the "present application." I currently hold the position of Vice President, Research and Development at Pentron Clinical Technologies, LLC. I have educational background in Dentistry and advanced degree in Dental Materials Science, and have been in the dental material field for about 25 years.

2. I have reviewed and am familiar with U.S. Patent No. 6,387,981 to Zhang et al., hereinafter "Zhang," which incorporates by reference U.S. Patent No. 6730,156 to Windisch et al., hereafter referred to as "Windisch," which patents were cited during the prosecution of the present application.

3. As described in my prior declaration of August 20, 2007, the independent claims in the present application claim nano-scaled silica particles in the form of chains. This type of silica is described in U.S. Patent No. 6,417,246, and can be purchased, for example,

from Nissan Chemical Industries, Ltd, under the trade name of SNOWTEX®-PS and SNOWTEX®-UP silica sol. This type of silica particle is nowhere mentioned or disclosed by Zhang/Windisch.

4. A description of the preparation of the nanocluster filler of Zhang/Windisch is described in column 25, lines 49-60, of Windisch. Windisch states, as follows:

> The fillers of this invention are preferably not fully densified. The term "fully dense," as used herein, is descriptive of a particle that is near theoretical density, having substantially no open porosity detectable by standard analytical techniques such as the B.E.T. nitrogen technique....the fillers of the invention are often manufactured in a process that includes heat treatment. The surface area of the filler after heat treatment is preferably quite high. The ratio of surface area after heat treatment compared to the surface area before heat treatment is preferably greater than about 50%, more preferably greater than about 80%.

5. In contrast to the above-quoted teachings of Zhang/Windisch, the density of the filler according to the presently claimed invention approaches the state of being fully densified, and the surface area of the filler contains substantially no open porosity.

6. The properties obtained by the presently claimed filler depend on the temperature of heat treatment. Windisch teaches heat treatment at a temperature of 550°C in the examples, as shown in col. 25, line 59, of the patent. In contrast, in Example 8 of the present application, heat treatments were carried out at temperatures of 800°C and 950°C.

7. Heat treatment at 550°C, as used in Windisch (or by reasonable extrapolation, at 600°C) would not produce the same results as heat treatment at about or above about 800C, as evidenced by experiments in which fillers, starting with the sols produced according to the present invention, were heat treated at different temperatures and then compared in terms of the density of the fillers (g/cm$^3$). It was estimated that a fully densified filler density is about 2.8 g/cm$^3$, based on heat treatment at 1100°C. The experimental results are shown in Table 1 below.

TABLE 1

| Heat Temperature (°C) | Dried and non-sintered filler (Dried @ about 100°C) | 550°C | 1100°C | Production (large scale) processed and heated @880°C |
|---|---|---|---|---|
| d1 | 2.49 | 2.63 | 2.76 | 2.79 |
| d2 | 2.29 | 2.57 | 2.87 | 2.75 |
| d(avg.) | 2.39 | 2.60 | 2.81 | 2.77 |

Additional experiments further differentiated the heat treatment used to prepare the Zhang/Windisch nanoclusters from the heat treatment used to prepare our processed fillers, in which surface area (SA) and porosity (Microvolume, MV), as determined by the BET method, were compared after heat treatment of the fillers at different temperatures were compared. The experimental results are shown in Table 2.

TABLE 2

| Temperature (°C) | Dried and non-sintered filler | 550°C | 1100°C | Production (large scale) processed and heated @880°C | Control* |
|---|---|---|---|---|---|
| SA (sq.m/g) | 111 | 109 | 0.17 | 41 | 15 |
| MV (cc/g) | 0.00056 | 0.00640 | 0.00002 | 0.00088 | 0.00090 |

*The control was regular barium-boro-silicate glass filler (Schott 8235, 0.7 um sized)

It is noted that the surface area of the present fillers decreased below the range specified by Windisch. It is further noted that the micropore volume at 550°C is substantially more than that for the non-heat processed starting material as well as the higher temperature heated materials according to the presently claimed invention.

8. In view of the above experiments, it can be concluded that the heat treatment of the filler of the present invention, starting with the elongated nanoparticles in the form of chains, produces a different product that differs in structure and properties from the nanoclusters produced according to the preparation of Windisch/Zhang, starting with monodispersed substantially spherical colloidal particles, after the required heat treatment.

9. It is further noted that heating at 800°C or above is believed to result in more crystallization than heating at 600°C since, as shown by Figure 1 in the present application, heat treatment at 800°C does, in fact, effectively cause crystallinity in the product, and it is reasonable for one of ordinary skill in the art to conclude that significantly greater crystallinity would be produced at 800°C than at 600°C.

10. In view of the above, the heat treatment of the elongated nanoparticles at a relatively higher temperature, in the presently claimed invention, results in different structure and properties in the final product, including, a product with significantly different crystallinity, density, surface area, and porosity.

11. As a result of the different structure and properties of the presently claimed filler, the product also exhibits improved strength for use in a dental composite, including improved fracture resistance, better wear resistance, compared to other products, containing silica particles, that are similar to the products disclosed by Zhang/Windisch. The better wear resistance of the material using the disclosed filler was illustrated in the Table 5.

I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the patent.

Date: 9/11/09

Weitao Jia